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IS 13704 (1993): Glycerol Esters of Wood Rosin (Ester Gums), Food Grade [FAD 8: Food Additives]

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काष्ठरॉजिन (इस्टरगम) के ग्लिसरॉल, खाद्य ग्रेड — विशिष्टि

Indian Standard

GLYCEROL ESTERS OF WOOD ROSIN (ESTER GUMS), FOOD GRADE — SPECIFICATION

UDC 664·099·6 : 547·426·1

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 1993

Price Group 2

AMENDMENT NO. 1 APRIL 2006
TO
IS 13704 : 1993 GLYCEROL ESTERS OF WOOD ROSIN (ESTER GUMS), FOOD GRADE — SPECIFICATION

(*Page* 1, *clause* 2) — Substitute '1699 : 1995 Methods of sampling and test for food colours (*second revision*)' *for* '1699 : 1974 Methods of sampling and test for food colours (*first revision*)'.

[*Page* 2, *Table* 1, *Sl No*. (iv), *col* 3] — Substitute '10' *for* '40'.

[*Page* 2, *Table* 1, *Sl No*. (v), *col* 3] — Substitute '2' *for* '3'.

[*Page* 2, *Table* 1, *Sl No*. (v), *col* 4] — Substitute '15 of IS 1699 : 1995' *for* '9 of IS 1699 : 1974'.

[*Page* 2, *Table* 1, *Sl No*. (vi), *col* 4] — Substitute '15 of IS 1699 : 1995' *for* '10 of IS 1699 : 1974'.

(*Page* 2, *clause* 5.1, *line* 5) — Substitute '4 of IS 1699 : 1995' *for* '3 of IS 1699 : 1974'.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JUNE 2008
TO
IS 13704 : 1993 GLYCEROL ESTERS OF WOOD ROSIN (ESTER GUMS), FOOD GRADE — SPECIFICATION

[*Page* 2, *Table* 1, *Sl No.* (v), *col* 2] — Substitute 'mg/kg' *for* 'mg kg'.

(FAD 8)

Reprography Unit, BIS, New Delhi, India

(FAD 8)

Reprography Unit, BIS, New Delhi, India

Food Additives Sectional Committee, FAD 8

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties and in some cases to enhance the nutritive values of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have strict quality control of these food additives. A series of standard is, therefore, being prepared by the Institution to cover purity and identification of these substances. These standards would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by the scientists, health authorities and international bodies.

Glycerol esters of wood rosin are produced by the esterification of pale wood rosin with food grade glycerin.

The use of glycerol esters of wood rosin has been permitted under the Prevention of Food Adulteration Rules for use as a masticatory substance in chewing gum base as well as a beverage stabilizer and clouding agent. When intended for use in chewing gum base, the product is usually purified by stream stripping, but when intended for use in adjusting the density of citric oils for beverages, it is purified by counter current steam distillation. This standard covers glycerol esters of wood rosin (ester gums) for use as beverage stabilizers and clouding agents.

In the preparation of this standard due consideration has been given to the provisions of the Prevention of Food Adulteration Act, 1954 and the rules framed thereunder and the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977*. However, this standard is subject to the restrictions imposed under these Acts wherever applicable.

In the preparation of this standard, considerable assistance has been derived from Food Chemical Codex, Pub. National Academy of Sciences and National Research Council, Washington, DC.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

GLYCEROL ESTERS OF WOOD ROSIN (ESTER GUMS), FOOD GRADE—SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for glycerol esters of wood rosin.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
548 (Part 1) : 1964	Methods of sampling and test for oils and fats : Part 1 Sampling, physical and chemical tests
1070 : 1992	Reagent grade water (*third revision*)
1699 : 1974	Methods of sampling and test for food colours (*first revision*)
11912 : 1986	Lime oil distilled, food grade

3 REQUIREMENTS

3.1 Description

Glycerol ester of wood rosin is a hard pale amber coloured resin produced by the esterification of pale wood rosin with food grade glycerin. It is soluble in acetone and benzene, insoluble in water.

3.2 Identification Test

3.2.1 The glycerol esters of wood rosin is identified by comparing its infra-red absorption spectrum with a typical spectum as shown in Fig. 1. The sample is melted and prepared for analysis on a potassium bromide plate.

3.3 The materials shall also conform to the requirements given in Table 1.

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be securely packed in well-filled containers with minimum access to light and moisture. The containers shall be such as to preclude contamination of the contents with metal or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

4.3 Marking

Each container shall be legibly marked to give the following information:

a) Name of the material including the words, food grade;

b) Source of manufacture;

c) Minimum net mass of the contents;

d) Batch or code number;

e) Date of manufacture; and

f) Any other details required as per the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977/ Prevention af Food Adulteration Rules, 1955.*

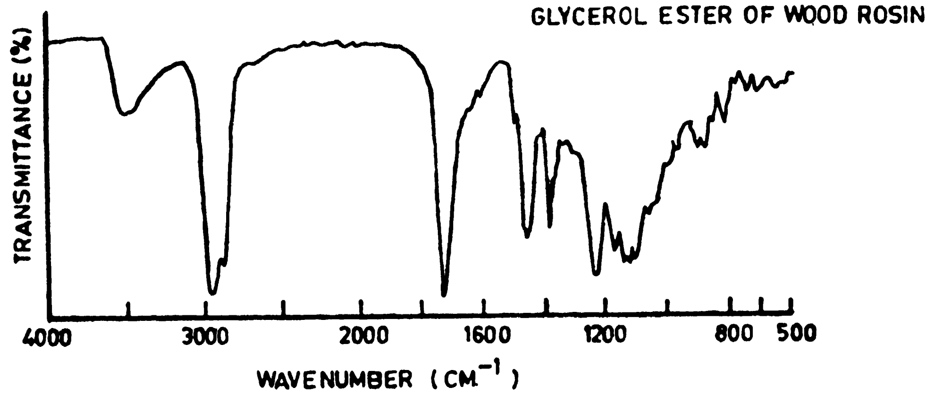


FIG. 1 INFRARED SPECTRA OF GLYCEROL ESTER OF WOOD ROSIN

Table 1 Requirements for Glycerol Ester of Wood Rosin
(*Clause* 3.3)

Sl No.	Characteristics	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Acid value (mg KOH/g)	3-9	Annex A of this standard
ii)	Drop softening point	88°-96°	Annex B of this standard
iii)	Hydroxyl number	15-45	16 of IS 548 (Part 1) : 1964
iv)	Heavy metals, mg/kg, *Max*	40	Annex B of IS 11912 : 1986
v)	Pb, mg kg, *Max*	3	9 of IS 1699 : 1974
vi)	As, mg/kg, *Max*	3	10 of IS 1699 : 1974

4.3.1 The container may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined according to the procedure prescribed in 3 of IS 1699 : 1974.

6 TESTS

6.1 Tests shall be carried out by the method as specified in 3.2 and col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

ANNEX A

[*Table* 1, *Sl No* (i)]

DETERMINATION OF ACID VALUE

A-1 PROCEDURE

A-1.1 Transfer about 4 grams of the sample, previously crushed into small lumps and accurately weighed, into a 250 ml Erlenmeyer flask. Add 50 to 75 ml of a 2 : 1 mixture of benzene : methanol, previously neutralized to phenolphthalein with sodium hydroxide. Dissolve the sample by shaking or heating gently, if necessary, then add about 0·5 ml of phenolphthalein and titrate with 0·5 N, alcoholic potassium hydroxide to the first pink colour that persists for 30 seconds.

A-2 CALCULATION

$$\text{Acid value} = 56{\cdot}1\, V \times \frac{N}{M}$$

where

V = the exact volume, in ml (accurate to 0·1 ml), of potassium hydroxide used;

N = exact normality (accurate to 0·01 N), of potassium hydroxide soluble; and

M = mass, in g, of the material taken for the test.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DROP SOFTENING POINT

B-1 DEFINITION

B-1.1 The drop softening point is defined as that temperature at which a given weight of rosin or rosin derivate begins to drop from the bulb of a special thermometer mounted in a test tube that is immersed in a constant-temperature bath.

B-2 APPARATUS

B-2.1 The apparatus illustrated in Fig. 2 consists of the components described in B-1.1 to B-2.1.3.

B-2.1.1 *Thermometer*

A special total-immersion softening point thermometer, covering the range from 0° to 250° and graduated in 1° divisions with bulb 16 ± 0·8 mm in length and 6·4 ± 0·14 mm in diameter.

B-2.1.2 *Heating Bath*

Use an 800- or 1 000-ml beaker containing a suitable heating medium. For rosins having a softening point above 80°, use glycerin, mineral oil or other suitable vegetable oil, depending upon the temperature range required. The temperature of the heating medium shall be maintained within ±1° of the temperature specified in Table 1. The medium shall be stirred constantly during the test with a suitable mechanical stirrer to ensure uniform heating of the medium.

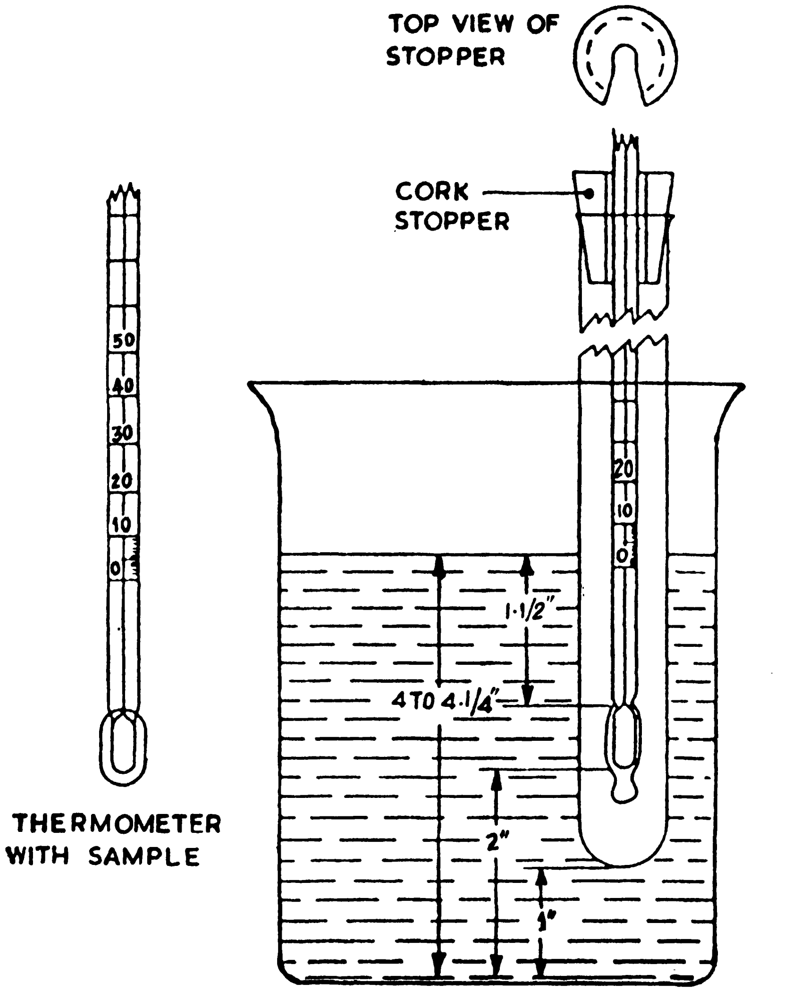
B-2.1.3 *Test Tube*

A standard 22 × 175-mm test tube with rim, fitted with a cork stopper as shown in Fig. 2.

B-3 PROCEDURE

B-3.1 Sample Preparation

Place about 20 g of the sample in a 50-ml beaker, and heat in an oven, on a sand bath or hot plate, or in an oil bath until its softening point is exceeded, but not more than 25° to 30° above it softening point or for any longer than necessary. Tare the softening point thermometer, and cautiously warm the bulb over a flame or hot plate until it registers 15° to 20° above the expected softening point of the sample. Immediately dip the thermometer bulb into the melted sample, withdraw, and rotate it so that a uniform film of the melted sample is deposited over the surface of the bulb, care being taken not to extend the film higher than the top of the bulb. Quickly place the thermometer on a balance, and weigh. The mass of the sample on the thermometer bulb should be between 500 and 550 mg. If the mass is low, again dip the bulb in the molten sample: if the



APPARATUS SHOWING APPEARANCE OF SAMPLE OF END POINT OF TEST

Fig. 2 Apparatus for Drop Softening Point Determination

mass is high, pull off some of the sample with the fingers. When the correct sample mass has been obtained, mould the sample uniformly around the bulb by rolling on the palm of the hand or between the fingers. The sample shall be of uniform thickness over the bulb, and it shall not extend up onto the thermometer stem (*see* Fig. 2). (If the film of the sample is not uniform when cooled, it should be completely removed from the bulb and a new one applied. Do not reheat the film and try to remould). Allow the film and thermometer to cool to room temperature, allowing about 15 minutes for cooling. (NOTE : If samples having high softening points crack or 'check' on the thermometer bulb upon cooling to room temperature, prepare sample film and cool only to about 50° below the expected softening point).

B-3.2 Procedure

Fill the glass beaker to a depth of not less than 100 mm or more than 108 mm with a suitable heating medium, support the beaker over a Bunsen burner, hot plate, or other suitable source of heat, and insert the bath stirrer and a bath temperature thermometer. Place the stirrer to one side so that the impeller clears the side of the beaker and is about 13 mm above the bottom of the beaker. Start the stirrer, heat the bath to the temperature specified in the monograph, and maintain this temperature within ± 1° throughout the test. Insert the prepared sample thermometer in the test tube, supporting it with a notched cork stopper so that the lower end of the bulb is 25 mm from the bottom of the test tube. Place the test tube in the bath so that the bottom of the thermometer bulb is 51 mm from the bottom of the beaker; the top of the bulb should be about 38 mm below the liquid level of the bath. Stir the bath in order to keep its temperature uniform throughout. Observe the sample thermometer, and record as the softening point the reading at which the elongated drop of sample on the end of the bulb first becomes constricted (*see* Fig. 2). Report the softening point to the nearest 0·5°.

B-3.2.1 *Precautions*

If the rosin crystallizes, making it difficult to obtain the correct softening point, prepare a new sample by heating the rosin rapidly, but cautiously, over a flame to a temperature of 160° to 170° in order to destroy all crystal nuclei; then dip the thermometer bulb into the molten rosin, remove momentarily, and rotate the thermometer to provide a uniform resin film on the bulb as it partially cools in air; dip the bulb in the melted sample repeatedly until the proper amount of resin has been deposited on the bulb. Results should not be reported if a crystal-free sample cannot be obtained.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'. Comments on this Indian Standard may be sent to BIS giving the following reference:

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